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BUREAU OF STANDARDS

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RECOMMENDED SPECIFICATIONS FOR QUICKLIME AND HYDRATED LIME FOR USE IN THE COOKING OF RAGS FOR THE MANUFACTURE OF PAPER

[This is the first of a series of specifications for the lime used in various chemical industries. To assist in the development of these specifications, the Bureau of Standards has called together an Interdepartmental Conference on Chemical Lime, composed of representatives of the Geological Survey and Bureau of Mines of the Interior Department; the Bureau of Soils, Bureau of Chemistry, and Forest Service of the Department of Agriculture; and the Nitrates Division of the War Department. The present specification, based on a draft originally prepared by F. A. Curtis, chief of the paper section, Bureau of Standards, has been unanimously approved by the above conference, and has also received the formal approval of the National Lime Association and the Technical Association of the Pulp and Paper Industry.]

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1. GENERAL

(*a*) USE OF LIME IN COOKING RAGS.—Rags are cooked in a digester under steam pressure, with lime, soda ash, or caustic soda, or a mixture of soda ash and lime. They are then washed to eliminate as much noncellulose material as possible.

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Either quicklime or hydrated lime may be used for cooking rags. The decision as to which to use is generally based on availability, cost, and present practice.

(*b*) DEFINITION OF QUICKLIME AND HYDRATED LIME.—Quicklime is the product resulting from the calcination of limestone. It will slake when water is added to it, and this slaking is accompanied by an evolution of heat and an increase in volume. It consists essentially of calcium oxide. Hydrated lime is a dry powder which is made by treating quicklime with enough water to satisfy its chemical affinity. It consists essentially of calcium hydroxide.

2. REQUIREMENTS

(*a*) QUALITY.—It is obvious that a clean lime, free from dirt and gritty substances, is desired.

(*b*) COMPOSITION.—Quicklime and hydrated lime, to meet the requirements of these specifications, shall not exceed the maximum percentages or fall below the minimum percentages given in the following table:

Ingredients	Quicklime		Hydrated lime	
	Maximum	Minimum	Maximum	Minimum
	Per cent	Per cent	Per cent	Per cent
Calcium oxide	.....	95	.....	72
Magnesium oxide	2	.....	1.6	.....
Oxides of silicon, iron, and aluminum, and other insoluble matter	2	.....	1.6	.....
Carbon dioxide	2	.....	1.6	.....

(*c*) PACKING.—Quicklime is shipped either in bulk in carload lots or barrels holding 180 pounds net or 280 pounds net each. Hydrated lime is shipped in paper bags holding 50 pounds net each.

(*d*) MARKING.—Each package, or each carload of bulk material, shall be legibly marked with the names of the consignor and consignee, and with some means of identifying the particular contract on which the shipment is made.

This information is in addition to that required by the Federal lime-barrel law.

3. SAMPLING AND TESTING

(*a*) SAMPLING.—The purchaser will bear all expense of sampling and testing. When quicklime is shipped in bulk, the sample shall be so taken that it will represent an average of all parts of the shipment from top to bottom, and shall not contain a disproportionate share of the top and bottom layers, which are most sub-

ject to changes. The sample shall comprise at least 10 shovelfuls taken from different parts of the shipment. The total sample taken shall weigh at least 100 pounds, and shall be crushed to pass a 1-inch ring, mixed thoroughly, and "quartered" to provide a 15-pound sample for the laboratory.

When quicklime is shipped in barrels, at least 3 per cent of the number of barrels shall be sampled. They shall be taken from various parts of the shipment, dumped, mixed, and sampled as specified in the above paragraph.

In the case of hydrated lime, the sample shall be a fair average of the shipment. Three per cent of the packages shall be sampled. The sample shall be taken from the surface to the center of the package. The material so obtained shall be thoroughly mixed and quartered to provide a 2-pound sample for the laboratory.

When sampling quicklime or hydrated lime, it is essential that the operation be conducted as expeditiously as possible, in order to avoid undue exposure of the material to the air. The sample to be sent to the laboratory shall immediately be placed in an air-tight container in which the unused portion shall be stored until the shipment has been finally accepted or rejected by the purchaser.

The sample may be taken either at the point of shipment or at the point of destination, as agreed upon by the contracting parties. The maximum limit for carbon dioxide as given in these specifications holds only when the sample is taken at point of shipment. If it is desired to take the sample at point of destination, due allowance must be made in order that the lime manufacturer shall not be penalized for the carbon dioxide absorbed in transit.

(*b*) TESTING.—The following directions are a brief summary of the analytical methods which are recommended. For more complete information on this subject reference should be made to The analysis of silicate and carbonate rocks, by W. F. Hillebrand, United States Geological Survey, Bulletin No. 700.

Blast 0.5 g of the sample for 15 minutes in a platinum crucible. Cool, and transfer to an evaporating dish. Mix to a slurry with distilled water. Add 5 to 10 cc concentrated HCl. Heat gently until solution is complete, breaking up lumps if necessary. Evaporate to dryness on water bath. Add 5 to 10 cc concentrated HCl, and dilute with an equal volume of distilled water. Digest on water bath for 10 minutes. Filter, and wash with hot water. Evaporate the filtrate to dryness. Dissolve in acid and water as before. Filter, and wash with hot water. Ignite the two precipitates together, and weigh as silica and insoluble matter.

Dilute the above filtrate to 250 cc. Add HCl if necessary to insure a total volume of 10 to 15 cc. Make alkaline with NH_4OH. Boil until odor of NH_3 is barely noticeable. Filter, and wash slightly with hot water. Dissolve the precipitate with hot dilute HCl, and repeat the precipitation as before. Filter, and wash thoroughly with hot water. Ignite, and weigh as oxides of iron and aluminum.

To the filtrates from the above add a few drops of NH_4OH and bring to a boil. Add 25 cc of a saturated solution of $(NH_4)_2C_2O_4$. Continue boiling until the precipitate becomes granular. Let stand until precipitate settles clear. Filter, and wash with boiling water. Ignite the precipitate, dissolve in dilute HCl, and dilute to 100 cc. Add excess of NH_4OH, and boil. Filter out any insoluble matter, ignite and weigh, and add its weight to the oxides of iron and alumina found previously. To this filtrate, add $(NH_4)_2C_2O_4$, proceeding as before. Filter, and wash with boiling water. Ignite and blast to constant weight as calcium oxide.

Acidify the filtrates from the above with HCl. Evaporate to 150 cc. Add 10 cc of a saturated solution of $NaNH_4HPO_4$ and boil. Cool. Add NH_4OH drop by drop, with constant stirring, until the precipitate starts to form. Then add moderate excess of NH_4OH. Stir for several minutes. Let stand overnight. Filter, and dissolve the precipitate in hot dilute HCl. Dilute to 100 cc, add 1 cc of saturated solution of $NaNH_4HPO_4$, and precipitate as before. Filter, and wash with an alkaline solution made by diluting NH_4OH until it contains about 2½ per cent NH_3 and then adding a few drops of HNO_3. Ignite, and weigh as $Mg_2P_2O_7$. Multiply this weight by $\frac{40}{111}$ to find the weight of MgO.

Place 5 g of the sample in a small Erlenmeyer flask and cover with hot distilled water. Connect this flask into a carbon-dioxide train, set up as follows: Next to the flask is a reflux condenser, to which is connected a calcium-chloride drying tube, followed by a tube containing anhydrous $CuSO_4$, then another tube of $CaCl_2$, then by two tubes filled with soda lime, and finally by another tube of $CaCl_2$. The entire train must be so arranged that a stream of CO_2 free air can be kept passing through it. Start this stream of air. Weigh the tubes containing soda lime and replace them in the train. Add to the sample in the flask about 25 cc of 1:1 HCl, being careful that no gas is lost and that the effervescence is not too violent during the operation. When

the effervescence diminishes, heat the flask, bringing the liquid gradually to boiling. Boil for 1 minute. Remove the flame and allow the flask to cool while the stream of air is still flowing, for 30 minutes. Disconnect and weigh the soda-lime tubes. Their increase in weight is recorded as carbon dioxide.

(*c*) RETESTING.—Notice of the rejection of a shipment based on these specifications must be in the hands of the consignor within 10 days after the receipt of the shipment at the point of destination. If the consignor desires a retest, he shall notify the consignee within 5 days of receipt of the notice of rejection. The original testing laboratory shall keep the sample sealed air-tight from the time they have taken out enough material for the original test until the expiration of the 15 days noted. The original testing laboratory shall agree to transmit the sample to any other laboratory for a retest at the direction of both of the contracting parties. This retest shall be at the expense of the consignor.

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